PA NT COOPERATION TREAT

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

Commissioner
US Department of Commerce
United States Patent and Trademark Office, PCT
2011 South Clark Place Room CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 13 March 2001 (13.03.01)	
International application No. PCT/FI00/00503	**Applicant's or agent's file reference** 49877
International filing date (day/month/year) 06 June 2000 (06.06.00)	**Priority date** (day/month/year) 07 June 1999 (07.06.99)
Applicant HÄNNINEN, Esko et al	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

29 December 2000 (29.12.00)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

[ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland**	Authorized officer Claudio Borton
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

Form PCT/IB/331 (July 1992)

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PCT REQUEST

49877

Original (for **SUBMISSION**) - printed on 06.06.2000 08:42:58 AM

0	**For rec iving Offic use only**	
0-1	International Application No.	PCT/FI 0 0 / 0 0 5 0 3
0-2	International Filing Date	0 6 JUN 2000 (0 6 -06- 2000)
0-3	Name of receiving Office and "PCT International Application"	The Finnish Patent Office PCT International Application
0-4	**Form - PCT/RO/101 PCT Request**	
0-4-1	Prepared using	PCT-EASY Version 2.90 (updated 10.05.2000)
0-5	**Petition** The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
0-6	**Receiving Office (specified by the applicant)**	National Board of Patents and Registration (Finland) (RO/FI)
0-7	**Applicant's or agent's file reference**	49877
I	**Title of invention**	METHOD FOR THE PREPARATION OF NICKEL CONCENTRATE
II	**Applicant**	
II-1	This person is:	applicant only
II-2	Applicant for	all designated States except US
II-4	Name	VALTION TEKNILLINEN TUTKIMUSKESKUS
II-5	Address:	Vuorimiehentie 5 FIN-02044 VTT Finland
II-6	State of nationality	FI
II-7	State of residence	FI
III-1	**Applicant and/or inventor**	
III-1-1	This person is:	applicant and inventor
III-1-2	Applicant for	US only
III-1-4	Name (LAST, First)	HÄNNINEN, Esko
III-1-5	Address:	Suorannantie 5 FIN-82500 Kitee Finland
III-1-6	State of nationality	FI
III-1-7	State of residence	FI

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III-2	**Applicant and/or inv nt r**	
III-2-1	This person is:	applicant and inventor
III-2-2	Applicant for	US only
III-2-4	Name (LAST, First)	LEPPINEN, Jaakko
III-2-5	Address:	Poikkikatu 11 FIN-83500 Outokumpu Finland
III-2-6	State of nationality	FI
III-2-7	State of residence	FI
III-3	**Applicant and/or inventor**	
III-3-1	This person is:	applicant and inventor
III-3-2	Applicant for	US only
III-3-4	Name (LAST, First)	HINTIKKA, Väinö
III-3-5	Address:	Kupariperä 6 FIN-83500 Outokumpu Finland
III-3-6	State of nationality	FI
III-3-7	State of residence	FI
IV-1	**Agent or common representative; or address for correspondence** The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:	agent
IV-1-1	Name	BERGGREN OY AB
IV-1-2	Address:	P.O. Box 16 FIN-00101 Helsinki Finland
IV-1-3	Telephone No.	+358-9-693701
IV-1-4	Facsimile No.	+358-9-6933944
IV-1-5	e-mail	email.box@berggren.fi
V	**Designation of States**	
V-1	Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT EP: AT BE CH&LI CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE and any other State which is a Contracting State of the European Patent Convention and of the PCT OA: BF BJ CF CG CI CM GA GN GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT

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V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AE AG AL AM AT AU AZ BA BB BG BR BY CA CH&LI CN CR CU CZ DE DK DM DZ EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US UZ VN YU ZA ZW
V-5	**Precautionary Designation Statement** In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.	
V-6	**Exclusion(s) from precautionary designations**	NONE
VI-1	**Priority claim of earlier national application**	
VI-1-1	Filing date	07 June 1999 (07.06.1999)
VI-1-2	Number	991294
VI-1-3	Country	FI
VI-2	**Priority document request** The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) identified above as item(s):	VI-1
VII-1	**International Searching Authority Chosen**	Swedish Patent Office (ISA/SE)
VIII	**Check list**	number of sheets / electronic file(s) attached
VIII-1	Request	4 / -
VIII-2	Description	9 / -
VIII-3	Claims	1 / -
VIII-4	Abstract	1 / 49877.txt
VIII-5	Drawings	1 / -
VIII-7	TOTAL	16
	Accompanying items	paper document(s) attached / electronic file(s) attached
VIII-8	Fee calculation sheet	✓ / -
VIII-9	Separate signed power of attorney	✓ / -
VIII-16	PCT-EASY diskette	- / diskette
VIII-17	Other (specified):	Copy of Official Action in FI 991294 / -
VIII-18	**Figure of the drawings which should accompany the abstract**	
VIII-19	**Language of filing of the international application**	Finnish

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IX-1	**Signature of applicant r ag nt**	
IX-1-1	Name	BERGGREN OY AB
IX-1-2	Name of signatory	Esko Heikkinen
IX-1-3	Capacity	Patent Agent

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10-1	**Date of actual receipt of the purported international application**	06 JUN 2000 (06-06-2000)
10-2	**Drawings:**	
10-2-1	Received	
10-2-2	Not received	
10-3	**Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application**	
10-4	**Date of timely receipt of the required corrections under PCT Article 11(2)**	
10-5	**International Searching Authority**	ISA/SE
10-6	**Transmittal of search copy delayed until search fee is paid**	

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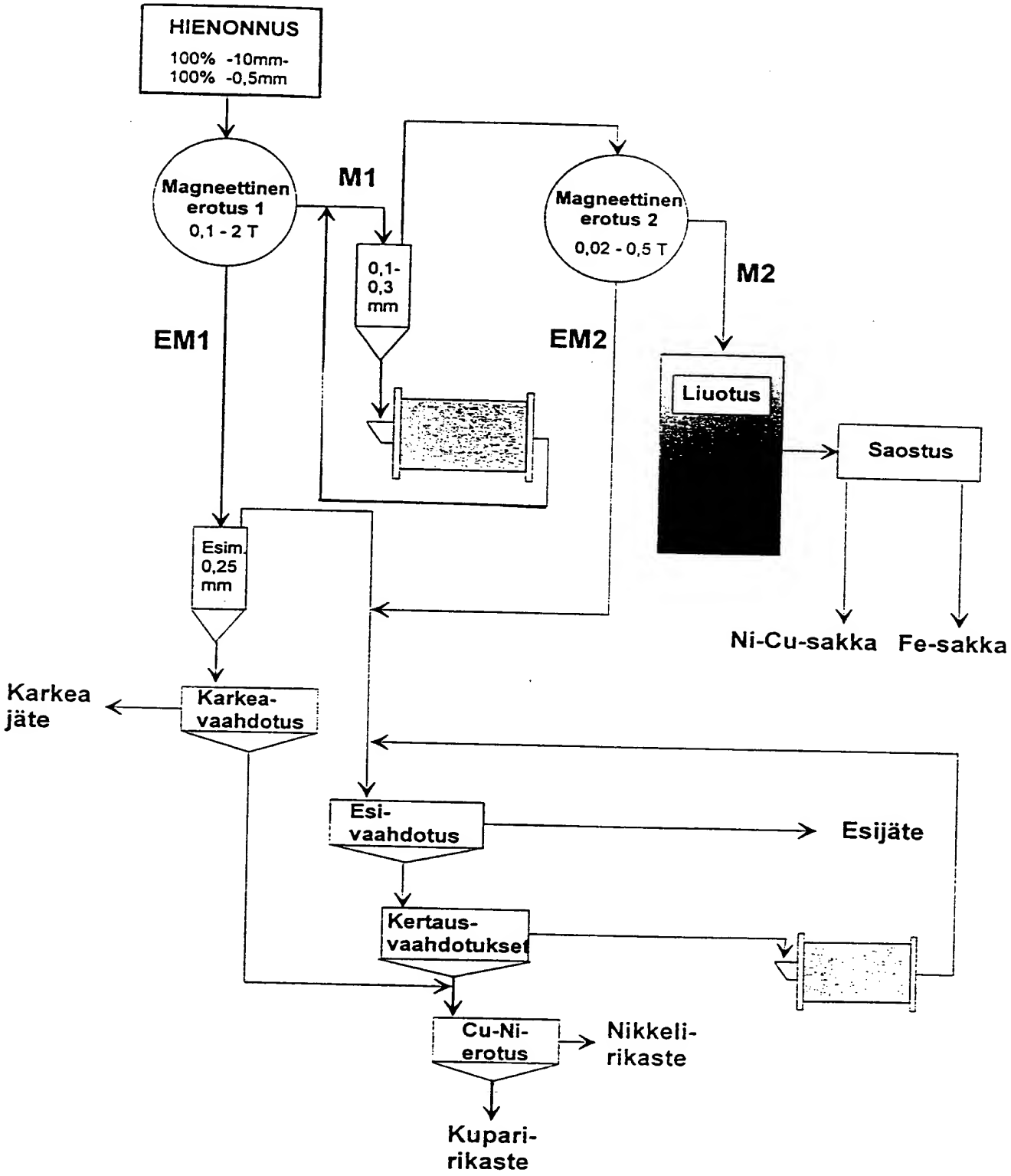
11-1	**Date of receipt of the record copy by the International Bureau**	23 JUNE 2000 (23.06.00)

KEKSINNÖN MUKAISEN PROSESSIN VIRTAUSKAAVIO



Nikkelirikasteen valmistusmenetelmä

Johdanto

Keksintö liittyy mineraalien rikastustekniikkaan ja koskee nikkelirikasteen valmistusmenetelmää (= nikkelimalmien rikastusprosessia).

Maapallon nikkelituotanto on pääosin peräisin magmaattissyntyisistä magneettikiisu-pentlandiitti-kuparikiisumalmeista, joissa silikaatit ja magneettikiisu ovat määrällisesti dominoivia mineraaleja. Arvosulfideja, pentlandiittia ja kuparikiisua, on vähän, useimmiten vain muutamia prosentteja. Näiden rikastamiseksi on perinteisesti noudatettu seuraavia metodeja:

- Materiaali on murskattu ja jauhettu vaahdotushienouteen, jolloin eri arvomineraalipartikkelit ovat pääosin omina erillisinä rakeina (puhtaaksi-jauhatusaste on jo tässä vaiheessa korkea). Tämän jälkeen on vaahdotettu yhteissulfidirikaste (Ni-pitoisuus 4-6 %). Kuparirikkaammista malmeista on joskus erotettu Cu-rikaste erikseen ennen nikkelivaahdotusta.

- Rikasteen Ni-pitoisuutta on saatu nousemaan (tasolle 6-10 %) ja määrää pienenemään painamalla magneettikiisua vaahdotuksessa jätteeseen. Saantitappiot ovat estäneet magneettikiisun kvantitatiivisen painamisen, sillä pentlandiitin ja magneettikiisun vaahdotusominaisuuksissa ei ole löydetty riittävän suurta eroa selektiivisen erotuksen onnistumiseksi.

- On suoritettu magneettikiisun magneettisia erotuksia prosessin eri vaiheiden tuotteille (syöte, esirikaste, rikaste), yleensä vaahdotushienouteen saatetulle materiaalille. Tämä on antanut kuitenkin vain osittaisen ratkaisun, koska kaikkea magneettikiisua ei saada poistettua magneettisesti tässä hienoudessa. Magneettikiisun eri faasit, monokliininen/ferromagneettinen ja heksagoninen /paramagneettinen, ovat jo tässä hienoudessa erillään toisistaan. Toisaalta magneettinen erotus ei ole riittävän selektiivinen hyvin hienoissa raeluokissa ja hienoja arvomineraaleja päätyy magneettiseen fraktioon. Nikkelin saantitappiot ovat olleet niin suuret, että magneettisesta erotuksestakaan ei ole tullut yleisesti käyttöönotettua menetelmää korkealaatuisten nikkelirikasteiden tuottamiseksi.

Keksinnön yleinen kuvaus

Nyt on keksitty patenttivaatimuksen 1 mukainen nikkelirikasteen valmistusmenetelmä. Muissa vaatimuksissa esitetään eräitä keksinnön edulliseen hyödyntämiseen liittyviä näkökohtia.

Menetelmä voi sisältää seuraavat vaiheet:

1. Mineraalien selektiivinen, asteittainen hienonnus prosessin eri vaiheissa

2. Magneettikiisun erotus magneettisin menetelmin

3. Karkeavaahdotus ja karkean silikaattifraktion poisto

4. Vaahdotus

5. Magneettikiisurikasteen liuotus ja arvometallien saostus

Menetelmän avulla saadaan hyvällä arvomineraalisaannilla nikkelipitoisuudeltaan korkeaa rikastetta perinteiseen nikkelirikastukseen verrattuna pienemmin investointi- ja käyttökustannuksin. Rikasteen laadun paranemisella on tuntuva ekonominen ja ekologinen merkityksensä myös valmistettujen rikasteiden jatkojalostusketjussa.

Keksinnön yksityiskohtainen kuvaus

Uusi menetelmä perustuu käsiteltävien mineraalien luonnollisen selektiivisen hienontuvuuden hyödyntämiseen käyttäen vanhoja, hyväksi koettuja rikastuskeinoja, luokitusta, magneettista erotusta ja vaahdotusta tavoitteena korkealaatuiset nikkeli- (ja kupari-) rikasteet mahdollisimman korkein arvometallisaannein. keksinnönmukainen hienonnus ja rikastusmenetelmien valinta ja niiden yhdistäminen uudella tavalla perustuu havaittuun arvo-mineraalien esiintymiseen hyödynnettävässä malmissa:

- Kyseisessä malmityypissä sulfidit esiintyvät keskenään yhteenkasvettuneina yleensä niitä huomattavasti kidekooltaan suurempien silikaattien välitiloissa. Pentlandiitti esiintyy aina ja kuparikiisu lähes aina magneettikiisun sisällä tai kyljessä.

- Pääosa pentlandiitista (70-80 %) esiintyy omamuotoisina kiteinä (Ø 0,3-20 mm), jotka ovat sisäisesti pirstoutuneet (Ø 0,01-0,3 mm). Alkujaan ehyt pentlandiittikide on alkuperäisessä asemassaan magneettikiisussa useimmiten kymmeninä pirstaleina. Pieni osa pentlandiitista (5-10 %) on pienikiteisinä (alle Ø 0,1 mm) raejonoina mag-

neettikiisukiteiden raja-pinnoilla ja pieni osa (5 %) suotaumaliekkeinä (alle Ø 0,02 mm) magneettikiisussa.

- Magneettikiisu on useissa malmeissa monokliinisen (ferromagneettisen) ja heksagonisen (paramagneettinen) faasin seos. Mineraali sisältää nikkeliä rautaa korvaavana keskimäärin 0,3-0,4 % (ns. hilanikkeli).

Perustuen edellä esitettyihin mineralogisiin seikkoihin ja käyttäen tukena jäljempänä esitettyjä alustavia, suuntaa-antavia koetuloksia esitetään korkealaatuisen nikkelirikasteen tuottamiseksi seuraavaa prosessia:

1. Mineraalien selektiivinen hienonnus

Hienomurskaus tai karkeajauhatus

Hienonnuksen tavoitteena on vapauttaa sulfidit silikaateista ja saattaa arvomineraalit pentlandiitti ja kuparikiisu vaahdotushienouteen mahdollisimman varhaisessa vaiheessa ylijauhautumisen minimoimiseksi. Sulfidien vapauttaminen silikaateista ei vaadi silikaattien hienontamista kidekokoaan hienommaksi. Esimerkkimalmilla tehdyissä kokeissa riitti ko. hienonnusvaiheen hienousasteeksi 100 % - 4 mm. Hienonnuslaitteeksi voidaan valita efektiivisin, taloudellinen, optimaalisesti raerajoja (karkea, kovempi silikaatti / pehmeämpi sulfidi) pitkin hienontava laite, joka hienontaa niin, että pehmyt sulfidiosue rikkoontuu (pentlandiittipirstaleet vapautuvat magneetti-kiisusta ja mahdollisimman pitkälle myös magneettikiisu vapautuu silikaateista), mutta silikaattikiteet ei välttämättä sanottavasti hienonnu. Merkittävä osa pentlandiitista ja kuparikiisusta vapautuu vaahdotushienouteen jo ko. hienonnusvaiheessa.

Vahvamagneettisen tuotteen jauhatus

Vahvamagneettisen erotuksen magneettisen tuotteen pitäisi sisältää kaikki vähänkin magneettikiisua sisältävät rakeet. Silloin päätyisi tähän tuotteeseen myös kaikki vapautumaton pentlandiitti ja valtaosa vapautumattomasta kuparikiisusta. Magneettisesta tuotteesta luokitetaan erilleen hieno ja karkea aines. Luokitusraja on malmityypistä riippuen välillä Ø 0,1-0,3 mm. Karkea osa johdetaan lisäjauhatukseen sekarakeiden särkemiseksi ja arvomineraalien vapauttamiseksi.

Vaahdotuspiirin kertausjätteen jauhatus

Vaahdotuspiirin syötteen karkeuden huomioiden on luonnollista, että kertausjätteisiin voi kumuloitua karkeita, lopulliseen rikasteeseen kelpaamattomia sekarakeita,

jotka voidaan hajottaa kevyellä lisäjauhatuksella ja palauttaa tuote sopivaan kohtaan vaahdotuspiiriä.

2. Magneettinen erotus

Vahvamagneettinen erotus

Primäärinen magneettierotus suoritetaan hienomurskeesta/karkeajauheesta, jolloin erotetaan kaikki magneettikiisua sisältävät rakeet magneettiseen tuotteeseen. Erotinlaitteen on oltava kenttävoimakkuudeltaan kulloisellekin tapaukselle riittävä. Monokliiniselle magneettikiisulle riittää heikompi kenttävoimakkuus, kun taas heksagoninen magneettikiisu vaatii erottuakseen magneettiseen tuotteeseen huomattavasti suuremman kenttävoimakkuuden. Vahvamagneettisen erotuksen magneettisesta tuotteesta erotetaan luokituksella hieno, puhdas magneettikiisu ja karkea, sekarakeinen magneettikiisu, joka lisäjauhetaan.

Heikkomagneettinen erotus

Erotus suoritetaan erotinlaitteella, joka erottaa magneettiseen tuotteeseen vain puhtaan magneettikiisun. Näin saadaan tuote, jonka nikkelipitoisuus on yleensä 0,8-1,0 %:n suuruusluokassa (sisältää magneettikiisussa olevan hilanikkelin ja pienen määrän pentlandiittipartikkeleita, jotka ovat suurimmaksi osaksi magneettikiisun sisällä olevia pieniä sulkeumia). Nikkelin saanti magneettiseen tuotteeseen on vastaavasti 10-15 %:n kertaluokkaa. Magneettierotuksen jäte (= ei-magneettinen tuote, joka sisältää silikaatteja, pentlandiittia ja kuparikiisua), on massaltaan pieni ja sisältää merkittävästi arvosulfideja. Se johdetaan yhdessä primäärierotuksen jätteen kanssa vaahdotuspiiriin.

3. Karkeavaahdotus ja karkean silikaattifraktion poisto

Vahvamagneettisen erotuksen jäte (pääosa syötteestä) luokitetaan käyttäen luokitusrajana maksimiraekokoa, jossa arvosulfidit vielä perinteisessä vaahdotusprosessissa kvantitatiivisesti vaahdottuvat (esim. Ø 0,25 mm). Luokituksen alitteesta vaahdotetaan karkeavaahdotustekniikalla arvosulfidit ja rikaste syötetään tuotteen laadun sanelemaan sopivaan prosessivaiheeseen varsinaisessa vaahdotuspiirissä. Vaahdotusjäte on karkeaa silikaattiainesta, joka toimitetaan joko jätealueelle tai hyötykäyttöön.

4. Vaahdotus

Vaahdotuksessa erotetaan arvosulfidit silikaateista omaksi (Ni-Cu-yhteisrikaste) tai omiksi rikasteikseen (Ni- ja Cu-rikasteet) normaalin sulfidivaahdotuksen olosuhteis-

sa. Kyseessä on pentlandiitin ja kuparikiisun vaahdottaminen eroon silikaateista ja mahdollisista muista sulfideista, kuten heksagonisesta magneettikiisusta ja pyriitistä. Esirikasteen kertauksissa pääasiallinen paino on silikaattien poistolla, jossa käytetään tunnettua tekniikkaa. Kertausjätteiden karkeapää voidaan palauttaa välijauhatukseen ja sieltä vaahdotukseen, jossa sekarakeisuus tulisi ilman välijauhatusta aiheut-tamaan kumuloituvaa kiertokuormaa kertauspiiriin ja myös saantitappioita.

5. Liuotus

Magneettikiisun poisto aiheuttaa noin 10-20 %:n saantitappiot nikkelin osalta. Magneettikiisufraktion nikkelipitoisuus on yleensä 0,8-1,5 %, edullisesti 0,8-1,0 %. Haluttaessa voidaan vielä ottaa talteen tähän tuotteeseen sisältyvä nikkeli liuotusteitse, esimerkiksi atmosfäärisellä happiliuotuksella, hapettavalla paineliuotuksella tai bakteeriliuotusmenetelmillä. Arvometallit saostetaan liuoksesta sopivaa menetelmää käyttäen, jolloin saadaan sakka (tai selektiivisaostuksen tapauksessa sakat), joka (jotka) voidaan jatkojalostaa esimerkiksi yhdessä rikasteiden kanssa.

Menetelmän tuottamat edut

Menetelmällä päästään alustavien, laboratoriomittakaavaisten, panoskokeiden tulosten perusteella arvioituna rikasteen nikkelipitoisuudessa 20 %:n tasolle, jolloin Nisaanti on 70-75 % tasolla. Lisäksi on liuotusteitse hyödynnettävissä magneettikiisurikasteen sisältämä nikkeli, joten kokonaissaantitappio on 10-15 %:n suurusluokassa tai alle sen. Rikasteiden määrät ovat tuntuvasti pienemmät verrattuna perinteisillä menetelmillä tuotettuihin rikasteisiin.

Prosessin hallinta yksinkertaistuu verrattuna perinteisiin nikkelirikastamoihin, kun vaahdotuspiirin massavirrat ovat tuntuvasti pienemmät magneettikiisu- ja silikaattipoiston ansiosta. Lisäjauhettavat tuotteet ovat massaltaan pieniä ja laadultaan suhteellisen homogeenisiä, jolloin jauhatusprosessi on paremmin kontrolloitavissa.

Tällä menetelmällä voidaan eliminoida myös monille nikkelimalmeille tunnusomainen ongelma, joka aiheutuu hienosta jauhatuksesta silikaattien läsnäollessa - keksinnön mukaisella menetelmällähän pääosa silikaateista poistetaan prosessista tuntuvasti karkeampana verrattuna perinteiseen. Perinteisellä koko materiaalin samanaikaisella hienojauhatuksella syntyy vaahdotusta vaikeuttavaa ja tuotteiden laatua huonontavaa yli hienoa (kolloidaalista) mineraaliainesta (liejua), joka samalla tekee vaahdotuspiirin monimutkaisemmaksi ja lisää kemikaalien kulutuksia (lisää prosessikustannuksia).

Rikastamon investointi- ja käyttökustannukset tulevat pienenemään keksinnön mukaisella menetelmällä mm. seuraavista syistä:

- Hienonnuslaitteisto ja hienonnusenergian käyttö voidaan minimoida.
- Vaahdotuspiiri voidaan tehdä laitteiltaan ja käytöltään normaalia pienemmäksi
- Rikasteen määrä pienenee, joten käsittelykustannukset (suodatus, kuivaus yms.) pienenevät.

Rikasteen laadun nousu ja määrän pieneneminen vaikuttavat merkittävästi jatkojalostusketjussa:

- Rahtikustannukset pienenevät
- Sulattokustannukset pienenevät
- Savukaasuista talteenotettavan rikin määrä pienenee

Oheisessa kuvassa esitetään esimerkkinä erään keksinnön mukaisen prosessin virtauskaavio.

Keksinnön mukaisella menetelmällä suoritettuja esimerkkikokeita

Malminäyte murskattiin leukamurskaimella ja hienonnettiin edelleen valssimurskaimella asteittain seuloen raekokoon -1,4 mm. Seula-analyysiä ei tehty. Jatkoprosessin tuotteiden seula-analyysistä voidaan päätellä, että materiaalista n. 60 % oli alle 0,25 mm (pentlandiitista n. 90 % ja kuparikiisusta n. 85 %). -0,032 mm fraktion osuus koko murskeesta on 20 %.

Magneettikiisu poistettiin vahvamagneettisella neorem-magneeteilla varustetulla rumpuerotuslaitteella (magneettikentän voimakkuus erotussolassa noin 0,1 T ja rummun pinnalla 0,3 T) märkäerotuksena. Magneettikiisun poisto onnistui likimain täydellisesti. Laskennallinen menetys ei-magneettiseen tuotteeseen oli 2,9 %.

Vahvamagneettisen erotuksen magneettisen tuotteen luokituksella (seula 0,125 mm) erotettiin lisäjauhatusta varten karkeat, magneettikiisupitoiset rakeet hienosta jo arvomineraaleista vapaasta (lukuunottamatta pentlandiittisuotautumia) magneettikiisusta. Karkean tuotteen lisäjauhatuksen tarkoituksena oli vapauttaa magneettikiisupartikkelien kylkiäisinä olleet pentlandiitti- ja kuparikiisurakeet. Tämän jälkeen tehtiin jauhetulla tuotteella magneettinen kertaus SALA:n heikkomagneettisella märkäerottimella.

Hieno, miltei arvomineraaleista vapaa magneettikiisu poistettiin omaksi tuotteekseen (magneettikiisurikaste). Laite oli SALA:n märkä, heikko-magneettinen rumpuerotin. Koe suoritettiin tekemällä magneettiselle tuotteelle lisäksi yksi kertauserotus (puhdistus) samalla erottimella.

Magneettikiisurikaste edellisestä kokeesta:

- magneettikiisupitoisuus oli 99 %
- nikkelipitoisuus oli 0,88 %
- magneettikiisun saanti oli 91 %
- nikkelin saanti oli 14 %
- kuparin saanti oli 7 %
- rakeisen pentlandiitin saanti oli 3 %

Yhdistetyillä magneettierotuksen ei-magneettisilla tuotteilla tehtiin vaahdotus. Vaahdotuksessa rikastettiin pentlandiitti ja kuparikiisu silikaattien jäädessä jätteeseen (osittain hyvinkin karkeana). Vaahdotuksen jätteeseen menivät myös magneettikiisu (vähäinen määrä heksagonista, magneettierotuksen läpäissyttä) ja pyriitti.

Esivaahdotuksessa, jossa pH-olosuhteet oli säädetty rikkihapolla (H_2SO_4) arvoon 6,5 malmin luontaisesta arvosta (9,0). Happoa kului 0,57 kg/t syötettä. Esivaahdotukseen lisättiin 300 g/t NaIBX (natriumisobutyyliksantaattia) ja 60 g/t vaahdotetta (Dowfroth 250). Tulos oli seuraava:

- rikasteen nikkelipitoisuus oli 8,1 % ja kuparipitoisuus 2,4 %
- sulfidipitoisuus oli 41 %
- saannit olivat: nikkeli 78,2 %, kupari 82,9 %, pentlandiitti 88 %
- nikkelitappiot jätteeseen olivat: +0,25 mm 4,1 %, 0,032-0,25 mm 1,5 %,- 0,032 mm 2,4 %

Esirikasteelle suoritettiin kaksi kertausta, joista ensimmäiseen lisättiin 100 g/t CMC:aa ja 50 g/t NaIBX:a. pH oli tässä kertausvaahdotuksessa 8,3-8,2. Toiseen kertausvaahdotukseen lisättiin 170 g/t soodaa (Na_2CO_3), jolloin lietteen pH-arvo kohosi 10,0:aan. Lisäksi annostettiin tähän vaiheeseen 50 g/t CMC:aa ja 75 g/t

NaIBX:a. Valmennusajat olivat kummassakin kertauksessa CMC:lle 5 min ja NaIBX:lle 2 min. Näin saatiin kahdesti kerrattu rikaste, jonka pääasialliset ominaisuudet olivat seuraavanlaiset:

- rikasteen nikkelipitoisuus oli 14,5 % ja kuparipitoisuus 4,3 %
- sulfidipitoisuus oli 74 %
- saannit olivat: nikkeli 72,9 %, kupari 77,5 %, pentlandiitti 82,1 %

Oheisessa taulukossa esitetään kokeen analyysit, mineraalipitoisuudet ja saannit.

Myöhemmässä kokeessa päästiin 1 %:n Ni-malmilla jo yhdellä kertausvaahdotuksella rikasteen Ni-pitoisuuteen 20,3 %. Tämä koe erosi ensimmäisestä kokeesta lähinnä siinä, että vaahdotuksen syöte oli Ni-pitoisuudeltaan runsas kaksinkertainen edelliseen verrattuna (1,48 % Ni/ 0,65 % Ni). Alkuperäisten malminäytteiden pitoisuus suhde oli vastaavasti 1,0 % Ni / 0,67 % Ni. Lisäksi erona oli esivaahdotuksen pH-arvo, joka jälkimmäisessä kokeessa oli 9,0-8,4; pH-säätäjänä $Ca(OH)_2$) ja esivaahdotuksessa käytetty sulfidikokooja kaliumbutyyliksantaatti KBX (100 g/t), edellisen kokeen NaIBX:n sijasta. Kokooja kemikaalit ovat käytännöllisesti katsoen samat, joten eron tuloksiin aiheuttivat lähinnä hieman rikkaampi malmi ja edullisemmat vaahdotusolosuhteet (muut sulfidit eivät vaahdottuneet rikasteeseen korkeamman pH-arvon ja pienemmmän kokooja-annostuksen takia). Näin päästiin edellä mainittuun parempaan tulokseen.

Analyysit, mineraalipitoisuudet, saannit

Tuote	Seulaluokka	Paino	Analyysit			Mineraalipitoisuudet					Saannit					Saannit piirissä		
	mm	%	Ni	Cu	S	NIP*	CUK	FEK	SK	MUUT	Ni	NIP	CUK	FEK	MUUT	NIP	CUK	FEK
Syöte	Bulk	100,0	0,635	0,177	5,12	1,64	0,51	10,8	0,1	87,0	100,0	100,0	100,0	100,0	100,0			
MR	Bulk	9,99	0,878	0,119	39,90	0,54	0,34	99,0		0,1	13,8	3,3	6,7	91,3	0,0			
ES	Bulk	90,02	0,608	0,184	1,26	1,77	0,53	1,0	0,2	96,7	86,2	96,8	93,3	8,0	100,1			
ER	Bulk	6,11	8,125	2,377	15,44	23,68	6,86	10,3	2,1	59,1	78,2	88,0	82,0	5,8				
Esivaahd.	0,500-1,000	18,81	0,070	0,050	0,38	0,20	0,14	0,5	0,1	99,0	2,1	2,2	5,3	0,9	21,4			
jäte	0,250-0,500	18,35	0,070	0,030	0,22	0,20	0,09	0,2	0,0	99,4	2,0	2,2	3,1	0,4	21,0			
	0,125-0,250	10,98	0,030	0,010	0,10	0,09	0,03	0,1	0,0	99,7	0,5	0,6	0,6	0,1	12,6	90,9	87,9	72,6
	0,063-0,125	11,13	0,030	0,009	0,09	0,09	0,03	0,1	0,0	99,8	0,5	0,6	0,6	0,1	12,8			
	0,032-0,063	9,67	0,030	0,010	0,09	0,09	0,03	0,1	0,0	99,8	0,5	0,5	0,5	0,1	11,1			
	-0,032	14,97	0,101	0,014	0,34	0,29	0,04	0,5	0,1	99,1	2,4	2,6	1,2	0,6	17,1			
	Bulk	83,91	0,060	0,024	0,229	0,17	0,07	0,4	0,0	99,4	8,0	8,7	11,3	2,9	95,8			
KR1	Bulk	3,88	12,45	3,66	23,27	36,30	10,56	15,0	3,0	35,1	76,1	85,7	80,1	5,4	1,6	97,3	97,7	92,3
KJ1	+0,125	0,41	1,300	0,286	3,26	3,75	0,83	3,4	0,7	91,3	0,8	0,9	0,7	0,1	0,4			
KJ1	-0,125	1,82	0,451	0,120	1,50	1,29	0,35	1,9	0,4	96,1	1,3	1,4	1,2	0,3	2,0			
	Bulk	2,23	0,607	0,151	1,82	1,74	0,43	2,2	0,4	95,2	2,1	2,4	1,9	0,4	2,4			
KR2	+0,125	0,90	11,800	3,000	21,40	34,42	8,66	13,8	2,8	40,3	16,7	18,8	15,2	1,2	0,4			
	-0,125	2,30	15,500	4,800	28,60	45,23	13,86	17,4	3,5	20,0	56,2	63,3	62,3	3,7	0,5			
	Bulk	3,20	14,46	4,29	26,58	42,19	12,40	16,4	3,3	25,7	72,9	82,1	77,5	4,9	0,9	95,9	96,8	90,3
KJ2	Bulk	0,68	2,970	0,659	7,69	8,56	1,90	8,3	1,7	79,6	3,2	3,5	2,5	0,5	0,6			

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NIP*= rakeinen pentlandiitti Magneettikiisun Ni=0,7 % sisältää hilanikkelin - 0,4 % - ja arvioitujen pentlandiittisuotautumien sisältämän nikkelin

Patenttivaatimukset

1. Menetelmä nikkelirikasteen valmistamiseksi magneettikiisu-pentlandiitti-malmista, tunnettu siitä, että siinä suoritetaan seuraavat vaiheet:

- malmi hienonnetaan siihen maksimiraekokoon, jossa pääosa sulfidimineraaleista vapautuu silikaatti- ja muista harmemineraaleista ja vapautuneet arvosulfidit ovat suoraan tai lisähienonnuksen kautta rikastettavissa korkealaatuisiksi lopputuotteiksi, joka maksimiraekoko on parhaiten välillä 100 % - 10 mm ... 100 % - 0,5 mm,

- hienonnetusta malmista erotetaan magneettisesti magneettikiisua sisältävät partikkelit magneettiseksi rikasteeksi (M1), jolloin jäljelle jää epämagneettinen tuote (EM1),

- haluttaessa magneettista rikastetta (M1) lisäjauhetaan ja erotetaan siitä magneettisen lisäerotuksen avulla magneettikiisu harmeesta ja arvosulfideista,

- magneettisen erotuksen jätteenä saadut epämagneettiset tuotteet (EM1, EM2) toimitetaan vaahdotukseen, jossa tuotetaan nikkeli- ja/tai muut arvosulfidirikasteet.

2. Vaatimuksen 1 mukainen menetelmä, jossa epämagneettisesta tuotteesta (EM1, EM2) poistetaan karkeaa ainesta ennen vaahdotusta.

3. Vaatimuksen 1 tai 2 mukainen menetelmä, jossa magneettista rikastetta (M1) lisäjauhetaan ja lisäjauhatusta siitä poistetaan hienoa ainesta, kuten alle 0,1-0,2 mm ainesta.

4. Jonkin vaatimuksen 1-3 mukainen menetelmä, jossa vaahdotuksessa suoritetaan ensin esirikastus karkeavaahdotustekniikkaa käyttäen.

5. Jonkin vaatimuksen 1-4 mukainen menetelmä, jossa vaahdotus suoritetaan kerraten ja jossa kertausjätteiden karkea pää palautetaan välijauhatukseen.

6. Jonkin vaatimuksen 1-5 mukainen menetelmä, jossa, magneettihienomurskeesta otetaan nikkeliä talteen liuottamalla.

(57) Tiivistelmä

Keksintö koskee menetelmä nikkelirikasteen valmistamiseksi magneettikiisu-pentlandiitti-malmista. Menetelmässä malmi hienonnetaan siihen maksimiraekokoon, jossa pääosa sulfidimineraaleista vapautuu silikaatti- ja muista harmemineraaleista ja vapautuneet arvosulfidit ovat suoraan tai lisähienonnuksen kautta rikastettavissa korkealaatuisiksi lopputuotteiksi, hienonnetusta malmista erotetaan magneettisesti magneettikiisua sisältävät partikkelit magneettiseksi rikasteeksi (M1). Magneettisen erotuksen jätteenä saadut epämagneettiset tuotteet (EM1, EM2) toimitetaan vaahdotukseen, jossa tuotetaan nikkeli- ja/tai muut arvosulfidirikasteet.

Search Report

PATENTTI- JA REKISTERIHALLITUS
Patentti- ja innovaatiolinja

TUTKIMUSRAPORTTI

PATENTTIHAKEMUS NRO	LUOKITUS
991294	B 03 B 9/00

TUTKITTU AINEISTO

Patenttijulkaisukokoelma (FI), tutkitut luokat

B03B, B03C, B03D

Tiedonhaut ja muu aineisto

EPO Documentation, EPO; World Patents Index, Derwent

VIITEJULKAISUT

Kategoria*)	Julkaisun tunnistetiedot	Koskee vaatimuksia
X	Joseph R. Boldt jr., The Winning of Nickel, Its Geology, Mining, and Extractive Metallurgy, Longmans Canada Limited, Toronto 1967, s. 222-224	1

* X Document of particular relevance when considered alone
Y Document of particular relevance when combining with one or more documents belonging to same category
A Document defining the general state of art

*) X Patentoitavuuden kannalta merkittävä julkaisu yksinään tarkasteltuna
Y Patentoitavuuden kannalta merkittävä julkaisu, kun otetaan huomioon tämä ja yksi tai useampi samaan kategoriaan kuuluva julkaisu
A Yleistä tekniikan tasoa edustava julkaisu, ei kuitenkaan patentoitavuuden este

Päiväys	Tutkija
24.1.2000	Pertti Helke

INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 00/00503

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: B03D 1/00 // B03D 103:02, B03C 1/00, C22B 23/00
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: B03D, B03C, C22B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Joseph R. Boldt, Jr., "The winning of Nickel its Geology, Mining, and Extractive Metallurgy", 1967, Methuen & Co. Ltd., (London), page 222, figure 22 --	1-6
X	US 4002463 A (ANTONIOS NESTORIDIS), 11 January 1977 (11.01.77), column 1, line 5 - line 14, claims 6,7,8, abstract --	1-6
X	GB 1064469 A (FUJI IRON & STEEL COMPANY LIMITED), 5 April 1967 (05.04.67), page 4, line 92 - line 103; page 4, line 125 - page 5, line 25; page 6, line 32 - line 44, claims 1-4 --	1-6

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" erlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
12 October 2000	17 -10- 2000

Name and mailing address of the ISA/	Authorized officer
Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. + 46 8 666 02 86	Mattias Arvidsson/ELY Telephone No. + 46 8 782 25 00

INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 00/00503

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3754896 A (IWAO IWASAKI), 28 August 1973 (28.08.73), column 2, line 15 - column 3, line 50, abstract --	1-6
A	JP 59052546 A (DOWA MINING CO LTD) 1984-03-27 (abstract) World Patents Index (online).London,U.K.: Derwent Publications, Ltd. (retrieved on 2000-10-11) Retrieved from: EPO WPI Database. DW9814, Accession No. 1984-111337 & JP 59052546 (DOUWA KOGYO KK) 1984-07-06 (abstract) (online)(retrieved on 2000-10-11). Retrieved from: EPO PAJ Database -- --------	1-6

INTERNATIONAL SEARCH REPORT

Information on patent family members

01/08/00

International application No.
PCT/FI 00/00503

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4002463 A	11/01/77	AU 507394 B	14/02/80
		AU 1256976 A	06/10/77
		BR 7602007 A	05/10/76
		CA 1076368 A	29/04/80
		CU 34489 A	08/09/78
		DE 2528137 A,B,C	21/10/76
		FI 760898 A	05/10/76
		FR 2306274 A,B	29/10/76
		GB 1539284 A	31/01/79
		JP 1073018 C	30/11/81
		JP 51122618 A	26/10/76
		JP 56014133 B	02/04/81
		NO 142790 B,C	07/07/80
		NO 760985 A	05/10/76
		PH 13308 A	06/03/80
		YU 56876 A	31/05/82
		ZA 7601693 A	30/03/77
GB 1064469 A	05/04/67	NONE	
US 3754896 A	28/08/73	NONE	

PATENT COOPERATION TREATY

REC'D 25 SEP 2001
WIPO PCT

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
49877/EH		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/FI00/00503	06/06/2000	07/06/1999
International Patent Classification (IPC) or national classification and IPC		
B03D1/00		
Applicant		
VALTION TEKNILLINEN TUTKIMUSKESKUS et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

   ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

   I ☒ Basis of the report
   II ☐ Priority
   III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   IV ☐ Lack of unity of invention
   V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   VI ☐ Certain documents cited
   VII ☐ Certain defects in the international application
   VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
29/12/2000	21.09.2001
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Tragoustis, M Telephone No. +49 89 2399 8623

Form PCT/IPEA/409 (cover sheet) (January 1994)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/FI00/00503

I. Basis of the report

1. With regard to the **elements** of the international application *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17))*:

Description, pages:

1-10 as originally filed

Claims, No.:

1-6 as originally filed

Drawings, sheets:

1/1 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	1-6
	No: Claims	
Inventive step (IS)	Yes: Claims	1-6
	No: Claims	
Industrial applicability (IA)	Yes: Claims	1-6
	No: Claims	

2. Citations and explanations
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

1. The application relates to a method for preparing nickel concentrate from pyrrhotite-pentlandite ore, which method is simple, easily controllable and results in high yields of nickel product.
The method steps defined in claim 1 and more specifically the step of separating the pyrrhotite particles from the comminuted ore by magnetic means as a magnetic concentrate followed by the step of subjecting the resulting magnetic concentrate to further magnetic separation in magnetic means of lower field intensity , wherein the two non magnetic products from the two magnetic separation steps are subjected to flotation in order to obtain further nickel concentrates cannot be seen in the documents cited in the Search Report.
US-A-4002463 discloses a process for upgrading nickel from nickel lateritic iron by firstly . The starting ore is in an oxide form and not a sulfide ore as in the present application. Magnetic separation is applied here as a final step after reduction roasting.
In GB-A-1064469 the treatment of nickeliferous oxide ore is described including roasting with a halide and magnetic separation of the roasted product.
The book of J. R. Boldt contains general information and does not disclose the claimed sequence of steps.
The other documents cited in the Search Report are also not pertinent to the claimed method.
Hence claim 1 and the dependent claims 2-6 meet the requirements of Art. 33 PCT.

2. Claim 1 is not clearly drafted (Art. 6 PCT).
The step of subjecting the first magnetic concentrate (M1) to a separation in second magnetic means of lower field intensity should be defined clearly (the last but one paragraph of present claim 1 is not clear: "separating... by means of additional separation").
The maximum grain size obtained in the first comminuting step should be defined clearly; it is an essential feature and hence the facultative word "preferably" should be removed.
It should be specified in claim 1 that the two non-magnetic products are obtained from the two magnetic separations.
The description (see page 2, lines 5-9) is not in line with claim 1.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
14 December 2000 (14.12.2000)

PCT

(10) International Publicati n Number
WO 00/74856 A1

(51) International Patent Classification[7]: B03D 1/00 // 103:02, B03C 1/00, C22B 23/00

(21) International Application Number: PCT/FI00/00503

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(25) Filing Language: Finnish

(26) Publication Language: English

(30) Priority Data:
991294 7 June 1999 (07.06.1999) FI

(71) Applicant *(for all designated States except US)*: **VALTION TEKNILLINEN TUTKIMUSKESKUS** [FI/FI]; Vuorimiehentie 5, FIN-02044 VTT (FI).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: **HÄNNINEN, Esko** [FI/FI]; Suorannantie 5, FIN-82500 Kitee (FI). **LEPPINEN, Jaakko** [FI/FI]; Poikkikatu 11, FIN-83500 Outokumpu (FI). **HINTIKKA, Väinö** [FI/FI]; Kupariperä 6, FIN-83500 Outokumpu (FI).

(74) Agent: **BERGGREN OY AB**; P.O. Box 16, FIN-00101 Helsinki (FI).

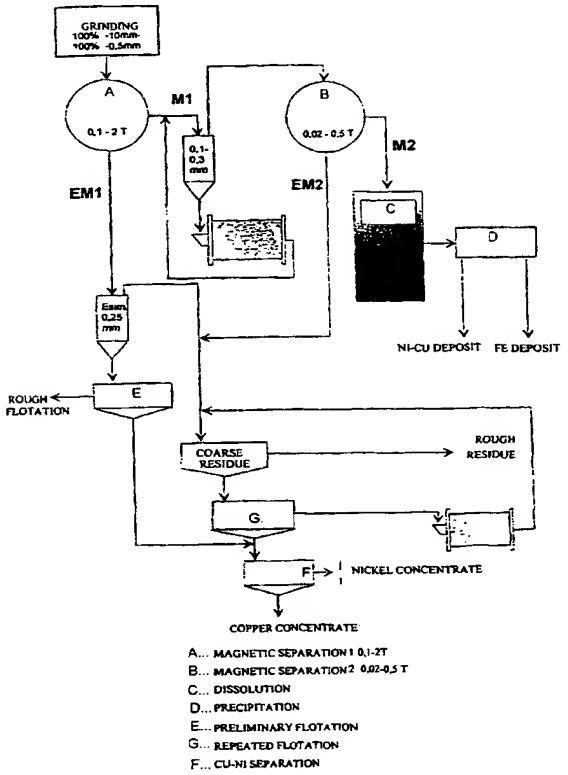
(81) Designated States *(national)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European

[Continued on next page]

(54) Title: METHOD FOR THE PREPARATION OF NICKEL CONCENTRATE

(57) Abstract: The invention relates to a method for preparing nickel concentrate from pyrrhotitepentlandite ore. The method comprises grinding the ore to the maximum grain size at which the major portion of the sulphide minerals is worked off from the silicate and other refuse minerals and the liberated precious sulphides can be concentrated to high-quality end products directly or by means of additional grinding, and separating the particles containing pyrrhotite magnetically from the ground ore to form a magnetic concentrate (M1). The non-magnetic products (EM1, EM2) obtained as a residue after magnetic separation are delivered to flotation, where the nickel and/or other precious sulphide concentrates are produced.



WO 00/74856 A1

patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

— *With international search report.*

— *Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.*

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Method for the preparation of nickel concentrate

Introduction

The invention relates to mineral concentrating technology and concerns a method for preparing nickel concentrates (= a process for concentrating nickel ores).

The major part of the global nickel production is derived from pyrrhotite-pentlandite-copper pyrite ore of magmatic origin, in which the quantitatively predominating minerals are silicates and pyrrhotite. The amounts of precious sulphides, pentlandite and pyrrhotite, are smaller, accounting only for a few per cent. The following methods have been conventionally implemented in the concentration of these minerals:

- crushing and grinding the material to flotation fineness, the different precious mineral particles being principally discrete grains (with a high degree of liberation at this early stage). This is followed by flotation of the conjugate sulphide concentrate (Ni content in the range of 4 to 6%). Before the nickel flotation, the Cu concentrate has occasionally been separated from the ores that are richest in copper.

- Increasing the Ni content of the concentrate (to a level in the range of 6 to 10%) and decreasing the amount by pressing pyrrhotite into the residue during flotation. The yield losses have prevented quantitative pyrrhotite pressing, because the difference found between pentlandite and pyrrhotite has not been sufficient for selective separation to be successful.

- Performing magnetic separation of pyrrhotite from the products in various process steps (feed material, rough concentrate, concentrate), usually from a material ground to flotation fineness. However, this has only yielded a partial solution, because pyrrhotite with this fineness will not be removed in its totality by magnetic means. The different pyrrhotite phases, monoclinic/ferromagnetic and hexagonal/-paramagnetic, are separated at this degree of fineness. On the other hand, in very fine grain size groups, magnetic separation is not sufficiently selective, and fine precious minerals will be present in the magnetic fraction. The losses of nickel yield have been so great that even magnetic separation has not become a commonly implemented method of producing high-quality nickel concentrates.

General description of the invention

The method for preparing nickel concentrates as defined in claim 1 has now been found. The other claims define a number of aspects of useful application of the invention.

The method may comprise the following steps:

1. Selective gradual comminution of the minerals in different process steps

2. Pyrrhotite separation by magnetic means

3. Rough flotation and removal of coarse fraction

4. Flotation

5. Dissolution of pyrrhotite concentrate and precipitation of precious metals

Compared with conventional nickel concentration, the method provides a preparation concentrate with higher nickel content and higher yield of precious minerals at lower investment and operating costs. The enhanced quality of the concentrate will also have an appreciable economic and ecologic impact on the further refining chain of the concentrates.

Detailed description of the invention

The new method is based on the utilisation of natural selective desintegration of the minerals to be prepared by using old, approved means of concentration, classification, magnetic separation and flotation aiming at high-quality nickel (and copper) concentrates with optimal yields of precious metals. The grinding of the invention and the choice and new combination of concentrating methods are based on the observed occurrence of precious minerals in the ore to be utilised:

- In this particular type of ore, sulphides occur in agglomerations in the interspaces between silicates having usually a notably greater crystal size than these. Pentlandite always occurs and copper pyrite nearly always occurs within or at the side of pyrrhotite.

- The major part of pentlandite (70 to 80%) occurs as idiomorphic crystals (Ø 0.3-20 mm), which are internally splintered (Ø 0.01-0.3 mm). An originally intact pentlandite crystal most frequently is splintered into dozens of fragments in its original position in pyrrhotite. A small portion of pentlandite (5-10%) form small-

crystal (under ∅ 0.1 mm) grain sequences on the interfaces between the pyrrhotite crystals and a small portion (5%) occur as filtering flames (under ∅ 0.02 mm) in the pyrrhotite.

- In most ores, pyrrhotite is a mixture of the monoclinic (ferromagnetic) and hexagonal (paramagnetic) phases. The mineral contains an average of 0.3 to 0.4% of nickel (so-called grid nickel) as an iron substitute.

Based on the mineralogical matters presented above and supported by the preliminary indicative test results given below, the following process is proposed for the production of high-quality nickel concentrates:

1. Selective mineral comminution

Fine crushing or coarse grinding

The purpose of comminution is to liberate sulphides from silicates and to grind the precious minerals pentlandite and pyrrhotite to flotation fineness at as early a stage as possible in order to minimise over-grinding. Liberating sulphides from silicates does not require the silicates to be ground under their crystal size. In the tests conducted with exemplifying ore, a degree of fineness of 100% - 4 mm in this comminuting step was enough. The choice of communitor may consist of the most efficient, economical device which performs optimal grinding following the grain limits (coarse, harder silicate/softer sulphide) such that the soft sulphide fraction is crushed (the pentlandite splinters are liberated from pyrrhotite and pyrrhotite is liberated from silicates as far as possible), but the silicate crystals are not necessarily reduced to a notable degree. A significant portion of pentlandite and pyrrhotite is liberated to flotation fineness already in this comminuting step.

Grinding of a highly magnetic product

The magnetic product of high-magnetic separation should contain all the grains containing pyrrhotite even in small amounts. In that case, all the unliberated pentlandite and the major portion of unliberated copper pyrite would end up in this product. The fine and coarse material of the magnetic product is separated into different groups. Depending on the type of ore, the classification limit is in the range ∅ 0.1 to 0.3 mm. The coarse fraction is led to further grinding in order to crush mixed grains and to liberate precious minerals.

Grinding of repeatedly prepared residue in the flotation circuit

Considering the coarseness of the material fed into the flotation circuit, coarse mixed grains which are unfit for use in the final concentrate may naturally cumulate in the repeatedly prepared residue, and these grains can be easily disintegrated with light additional grinding and the product can be returned to a suitable point of the flotation circuit.

2. Magnetic separation

High-magnetic separation

Primary magnetic separation is carried out from fine metal/coarse powder, all the grains containing pyrrhotite being separated to the magnetic product. The separator should have adequate field intensity for each individual case. Lower field intensity will be enough for monoclinic pyrrhotite, whereas hexagonal pyrrhotite requires considerably stronger field intensity in order to separate into the magnetic product. From the magnetic product of high-magnetic separation, a fine, pure pyrrhotite and a coarse pyrrhotite with mixed grains are separated by classification, the latter being further ground.

Low-magnetic separation

The separation is performed with a separator, which separates only pure pyrrhotite into the magnetic product. This yields a product with a nickel content of the usual order of 0.8 to 1.0% (containing the grid nickel in the pyrrhotite and a small amount of pentlandite particles, which are mainly small xenoliths within the pyrrhotite). The nickel yield in the magnetic product is accordingly of the order of 10-15%. The magnetic separation residue (= a non-magnetic product containing silicates, pentlandite and pyrrhotite) has a small volume and contains precious sulphides in a significant amount. It is conducted to the flotation circuit along with the primary separation residue.

3. Rough flotation and removal of the coarse silicate fraction

The high-magnetic separation residue (the major portion of the feed material) is classified using as a classification limit the maximum grain size in which precious sulphides are still quantitatively flotated (e.g. ∅ 0.25 mm) in a conventional flotation +process). From the fraction below classification, precious sulphides are flotated with rough-flotation techniques and the concentrate is fed into the suitable

process step of the actual flotation circuit according to the product quality. The flotation residue is a coarse silicate material, which is either taken to a dump or reclaimed.

4. Flotation

In flotation, precious sulphides are separated from silicates as a separate concentrate (Ni-Cu conjugate concentrate) or separate concentrates (Ni and Cu concentrates) in normal sulphide flotation conditions. The flotation aims at separating pentlandite and copper pyrite from silicates and any other sulphides present, such as hexagonal pyrrhotite and pyrite. The repeated preparation of rough concentrate focuses on silicate removal using conventional techniques. The coarse end of repeatedly prepared residues can be returned to intermediate grinding and from there to flotation, where the mixed grains would produce a cumulative circulating load on the repetitive circuit and would also entail losses of yield if no intermediate grinding were performed.

5. Dissolution

Pyrrhotite removal causes yield losses of approx. 10 to 20% regarding nickel. The pyrrhotite fraction usually has a nickel content of 0.8 to 1.5%, preferably 0.8 to 1.0%. If desired, the nickel contained in this product can be further recovered by dissolution, for instance atmospheric oxygen dissolution, oxygenating pressure dissolution or bacterial dissolution methods. Precious minerals are precipitated from the solution with a suitable method, resulting in a deposit (or deposits in the case of selective precipitation), which can be further refined jointly with the concentrates, for instance.

Benefits gained by the method

Estimated on the results of preliminary batch tests on laboratory scale, the method yields a 20% nickel content in the concentrate, the Ni yield being at the level of 70 to 75%. In addition, the nickel contained in the pyrrhotite can be utilised by dissolution, so that the overall yield loss will be of the order of 10 to 15% or less. The concentrate amounts are notably smaller than those produced with conventional methods.

The process control is simpler than at conventional nickel concentrating plants, because the mass flows in the flotation circuit are notably smaller owing to the

pyrrhotite and silicate removal. The products to be further ground have a small mass and relatively homogenous quality, thus allowing better process control.

This method also allows the elimination of the typical problem of many nickel ores, which is caused by fine grinding in the presence of silicates, given that the method of the invention removes a major portion of the silicates from the process in a notably coarser form than in conventional processes. The conventional simultaneous fine grinding of the total material produces over ground (colloidal) mineral material (sludge) which has a negative effect on the flotation and the quality of the products, and also calls for a more complex flotation circuit and increased chemical consumption (i.e. higher production costs).

The investment and operating costs of concentrating plants will diminish with the method of the invention i.a. for the following reasons:

- Minimised comminuting apparatus and use of grinding energy,

- Smaller-sized apparatus and reduced use of the flotation circuit compared to conventional flotation circuits

- A decrease in the concentrate amount, resulting in reduced processing costs (filtering, drying etc.).

The higher concentrate quality and smaller concentrate amount has a significant bearing on the further refining chain:

- Lower freight charges

- Lower smelting plant costs

- Smaller amount of sulphur to be recovered from flue gases

The enclosed figure is an exemplifying flow diagram of a process in accordance with the invention.

Exemplifying tests conducted with the method of the invention

An ore sample was crushed with a jaw crusher and was further comminuted with a roll crusher during gradual screening to a grain size of -1.4 mm. No screen analysis was performed. The screen analysis of the products of the further process allows the conclusion that approx. 60% of the material was under 0.25 mm (approx. 90% of

pentlandite and approx. 85% of copper pyrite). The - 0.032 mm fraction accounted for 20% of the total amount of crushed product.

The pyrrhotite was removed with a drum separator equipped with high-magnetic neorem magnets (magnet field intensity of approx. 0.1 T in the separation duct and of 0.3 T on the drum surface) in wet separation. The removal of pyrrhotite was almost totally successful. The calculatory loss to the non-magnetic product was 2.9%.

Using classification (0.125 mm mesh) of the magnetic product of high-magnetic separation, the coarse grains containing pyrrhotite were separated for further grinding from the fine pyrrhotite, which was already free from precious minerals (with the exception of pentlandite filtering products). The purpose of further grinding of the coarse product was to liberate the pentlandite and copper pyrite grains accompanying the pyrrhotite particles. After this magnetic repeated preparation was performed with the ground product using a SALA low-magnetic wet separator.

The fine pyrrhotite, which was almost free of precious minerals, was removed as a separate product (pyrrhotite concentrate). The apparatus was a SALA low-magnetic wet drum separator. The test was conducted by subjecting the magnetic product to an additional iterative separation (purification) with the same separator.

The pyrrhotite concentrate from the preceding test had

- A pyrrhotite content of 99%
- A nickel content of 0.88%
- A pyrrhotite yield of 91%
- A nickel yield of 14%
- A copper yield of 7%
- A yield of granular pentlandite of 3%

Flotation was performed with the combined non-magnetic products of the magnetic separation. During the flotation, pentlandite and pyrrhotite were concentrated while the silicates were left in the residue (partly even in quite a coarse form). The

flotation residue also comprised the pyrrhotite (of which a small amount was hexagonal and had been subjected to magnetic separation) and pyrite.

Preliminary flotation, in which the pH conditions were controlled with sulphuric acid (H_2SO_4) to a value of 6.5 of the natural ore value (9.0). The acid consumption was 0.57 kg/t of material feed. 300 g/t of NaIBX (sodium isobutyl xanthate) and 60 g/t of frothing agent (Dow froth 250) were added to the preliminary flotation. The result was:

- A concentrate with a nickel content of 8.1% and a copper content of 2.4%

- A sulphide content of 41%

- Yields: 78.2% of nickel, 82.9% of copper, 88% of pentlandite

- Nickel losses in the residue: +0.25 mm 4.1%, 0.032-0.25 mm 1.5%-0. 032 mm 2.4%

The rough concentrate was subjected to two iterative preparations, with additions of 100 g/t of CMC and 50 g/t of NaIBX to the first one. In this iterative flotation, the pH range was 8.3 to 8.2. 170 g/t of soda (Na_2CO_3) was added to the second iterative flotation, whereby the pH value of the sludge rose to 10.0. In addition, 50 g/t of CMC and 75 g/t of NaIBX were batched in this step. In both the iterative preparations, the preparation periods were 5 minutes for CMC and 2 minutes for NaIBX. Thus a concentrate that had been prepared twice was obtained, whose main properties were:

- A concentrate with a nickel content of 14.5% and a copper content of 4.3%

- A sulphide content of 74%

- Yields: 72.9% of nickel, 77.5% of copper, 82.1% of pentlandite

The enclosed table shows the test analyses, mineral contents and yields.

An ulterior test achieved a Ni content of 20.3% of the concentrate with 1% Ni ore and one single iterative flotation. This test differed from the first test mainly in that the flotation feed material was rich in Ni, with a double Ni content compared to the preceding one (1.48% of Ni/0.65% of Ni). The content ratio of the original ore samples was accordingly 1.0 % of Ni/0.67% of Ni. A further difference was the pH value of the rough flotation, i.e. in the range from 9.0 to 8.4 in the latter test; the pH

regulating agent was $Ca(OH)_2$ and the sulphide collector used in the preliminary flotation was potassium butyl xanthate KBX (100 g/t), instead of NaIBX as in the preceding test. The collecting chemicals are practically the same, so that the different results were chiefly brought about by the slightly richer ore and the more advantageous flotating conditions (the other sulphides were not flotated in the concentrate due to the higher pH value and the smaller collecting chemical batching). Thus the better result mentioned above was achieved.

Analyses, mineral contents, yields

Product	Screen class	Weight	Analyses			Mineral contents					Yields					Yields in the circuit		
	mm	%	Ni	Cu	S	NIP*	CUK	FEK	SK	OTHERS	Ni	NIP	CUK	FEK	OTHERS	NIP	CUK	FEK
Feed material	Bulk	100.0	0.635	0.177	5.12	1.64	0.51	10.8	0.1	87.0	100.0	100.0	100.0	100.0	100.0			
MR	Bulk	9.99	0.878	0.119	39.90	0.54	0.34	99.0		0.1	13.8	3.3	6.7	91.3	0.0			
ES	Bulk	90.02	0.608	0.184	1.26	1.77	0.53	1.0	0.2	96.7	86.2	96.8	93.3	8.0	100.1			
ER	Bulk	6.11	8.125	2.377	15.44	23.68	6.86	10.3	2.1	59.1	78.2	88.0	82.0	5.8				
Rough flot.	0.500-1.000	18.81	0.070	0.050	0.38	0.20	0.14	0.5	0.1	99.0	2.1	2.2	5.3	0.9	21.4			
residue	0.250-0.500	18.35	0.070	0.030	0.22	0.20	0.09	0.2	0.0	99.4	2.0	2.2	3.1	0.4	21.0			
	0.125-0.250	10.98	0.030	0.010	0.10	0.09	0.03	0.1	0.0	99.7	0.5	0.6	0.6	0.1	12.6	90.9	87.9	72.6
	0.063-0.125	11.13	0.030	0.009	0.09	0.09	0.03	0.1	0.0	99.8	0.5	0.6	0.6	0.1	12.8			
	0.032-0.063	9.67	0.030	0.010	0.09	0.09	0.03	0.1	0.0	99.8	0.5	0.5	0.5	0.1	11.1			
	-0.032	14.97	0.101	0.014	0.34	0.29	0.04	0.5	0.1	99.1	2.4	2.6	1.2	0.6	17.1			
	Bulk	83.91	0.060	0.024	0.229	0.17	0.07	0.4	0.0	99.4	8.0	8.7	11.3	2.9	95.8			
KR1	Bulk	3.88	12.45	3.66	23.27	36.30	10.56	15.0	3.0	35.1	76.1	85.7	80.1	5.4	1.6	97.3	97.7	92.3
KJ1	+0.125	0.41	1.300	0.286	3.26	3.75	0.83	3.4	0.7	91.3	0.8	0.9	0.7	0.1	0.4			
KJ1	-0.125	1.82	0.451	0.120	1.50	1.29	0.35	1.9	0.4	96.1	1.3	1.4	1.2	0.3	2.0			
	Bulk	2.23	0.607	0.151	1.82	1.74	0.43	2.2	0.4	95.2	2.1	2.4	1.9	0.4	2.4			
KR2	+0.125	0.90	11.800	3.000	21.40	34.42	8.66	13.8	2.8	40.3	16.7	18.8	15.2	1.2	0.4			
	-0.125	2.30	15.500	4.800	28.60	45.23	13.86	17.4	3.5	20.0	56.2	63.3	62.3	3.7	0.5			
	Bulk	3.20	14.46	4.29	26.58	42.19	12.40	16.4	3.3	25.7	72.9	82.1	77.5	4.9	0.9	95.9	96.8	90.3
KJ2	Bulk	0.68	2.970	0.659	7.69	8.56	1.90	8.3	1.7	79.6	3.2	3.5	2.5	0.5	0.6			

NIP*= granular pentlandite Pyrrhotite Ni=0.7 % contains grid nickel – 0.4 % - and the nickel contained in the estimated pentlandite filtrates

Claims

1. A method for preparing a nickel concentrate from pyrrhotite-pentlandite ore, characterised in that the following steps are performed:

- comminuting the ore to the maximum grain size at which the major portion of the sulphide minerals is liberated from silicate and other refuse minerals and the liberated precious sulphides can be concentrated to high-quality end products directly or by means of additional grinding, the maximum grain size being preferably in the range 100% - 10 mm...100% - 0.5 mm,

- separating by magnetic means from the comminuted ore the particles containing pyrrhotite as a magnetic concentrate (M1), the resulting residue being a non-magnetic product (EM1),

- if desired, additional grinding of the magnetic concentrate (M1) and separating the pyrrhotite from the refuse minerals and precious sulphides by means of additional separation,

- delivering the non-magnetic products (EM1, EM2) obtained as a residue after the magnetic separation to flotation, where nickel and/or other precious sulphide concentrates are produced.

2. A method as defined in claim 1, in which coarse material is removed from the non-magnetic products (EM1, EM2) before flotation.

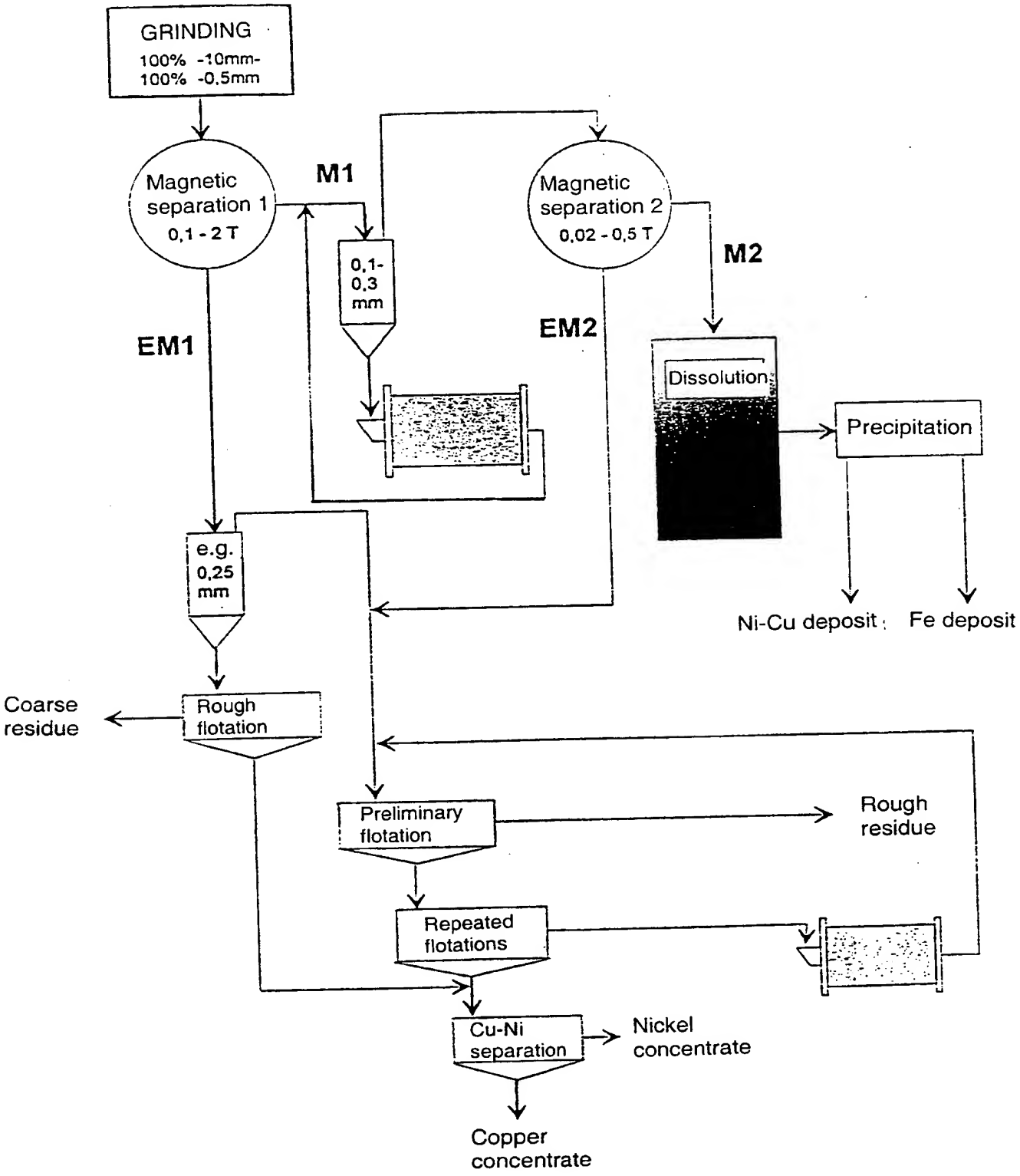
3. A method as defined in claim 1 or 2, in which the magnetic concentrate (M1) is additionally ground, and during the additional grinding fine material such as material under 0.1 to 0.2 mm is removed.

4. A method as defined in any of claims 1 to 3, in which rough concentration is first carried out during the flotation by means of rough flotation techniques.

5. A method as defined in any of claims 1 to 4, in which the flotation is carried out repeatedly and in which the coarse end of the repeatedly prepared refuse is returned to intermediate grinding.

6. A method as defined in any of claims 1 to 5, in which nickel is recovered from the magnetic fine crushed product by means of dissolution.

FLOW DIAGRAM OF THE PROCESS OF THE INVENTION



INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 00/00503

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: B03D 1/00 // B03D 103:02, B03C 1/00, C22B 23/00
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: B03D, B03C, C22B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Joseph R. Boldt, Jr., "The winning of Nickel its Geology, Mining, and Extractive Metallurgy", 1967, Methuen & Co. Ltd., (London), page 222, figure 22 --	1-6
X	US 4002463 A (ANTONIOS NESTORIDIS), 11 January 1977 (11.01.77), column 1, line 5 - line 14, claims 6,7,8, abstract --	1-6
X	GB 1064469 A (FUJI IRON & STEEL COMPANY LIMITED), 5 April 1967 (05.04.67), page 4, line 92 - line 103; page 4, line 125 - page 5, line 25; page 6, line 32 - line 44, claims 1-4 --	1-6

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" erlier document but published on or after the international filing date
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"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
12 October 2000	17 -10- 2000
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. + 46 8 666 02 86	Authorized officer Mattias Arvidsson/ELY Telephone No. + 46 8 782 25 00

INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 00/00503

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3754896 A (IWAO IWASAKI), 28 August 1973 (28.08.73), column 2, line 15 - column 3, line 50, abstract ---	1-6
A	JP 59052546 A (DOWA MINING CO LTD) 1984-03-27 (abstract) World Patents Index (online).London,U.K.: Derwent Publications, Ltd. (retrieved on 2000-10-11) Retrieved from: EPO WPI Database. DW9814, Accession No. 1984-111337 & JP 59052546 (DOUWA KOGYO KK) 1984-07-06 (abstract) (online)(retrieved on 2000-10-11). Retrieved from: EPO PAJ Database --- --------	1-6

INTERNATIONAL SEARCH REPORT

Information on patent family members

01/08/00

International application No.
PCT/FI 00/00503

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4002463 A	11/01/77	AU 507394 B	14/02/80
		AU 1256976 A	06/10/77
		BR 7602007 A	05/10/76
		CA 1076368 A	29/04/80
		CU 34489 A	08/09/78
		DE 2528137 A,B,C	21/10/76
		FI 760898 A	05/10/76
		FR 2306274 A,B	29/10/76
		GB 1539284 A	31/01/79
		JP 1073018 C	30/11/81
		JP 51122618 A	26/10/76
		JP 56014133 B	02/04/81
		NO 142790 B,C	07/07/80
		NO 760985 A	05/10/76
		PH 13308 A	06/03/80
		YU 56876 A	31/05/82
		ZA 7601693 A	30/03/77
GB 1064469 A	05/04/67	NONE	
US 3754896 A	28/08/73	NONE	